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RESEARCH MEMORANDUM

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PROPERTIES OF FUMING NITRIC ACIDS

By Charles E. Feiler and Gerald Morrell

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NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
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INVESTIGATION OF EFFECTS OF ADDITIVES ON STORAGE PROPERTIES

OF FUMING NITRIC ACIDS

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SUMMARY

The storage properties of fuming nitric acids, with and without additives, were studied at a temperature of 170° F in closed containers of approximately 100-milliliter capacity; the containers had aluminum bodies and stainless steel caps. It was found that at equal ullages the peak decomposition pressures developed by white fuming nitric acid (WFNA) are of an order of magnitude greater than the peak pressures developed by red fuming nitric acid (RFNA) containing approximately 19 percent nitrogen dioxide and 3 percent water. Similar results have been reported by other investigators.

The effects on storage properties of 1 percent additions of several materials, including potassium nitrate, 85-percent phosphoric acid, sodium fluoride, and potassium fluoride dihydrate, were investigated and the data indicate that, of this group, the potassium fluoride dihydrate is most effective in reducing the rate of pressure rise and in reducing corrosion on aluminum and stainless steel surfaces. An addition of 1 percent of potassium fluoride dihydrate to white fuming nitric acid and red fuming nitric acid had no effect on the ignition delay of a representative fuel at 70° F even after 100 hours of storage at 170° F in an aluminum container.

INTRODUCTION

At temperatures of the order of 150° F and higher, highly concentrated nitric acid undergoes appreciable decomposition according to the following equation:

$$2HNO_3 \rightleftharpoons 2NO_2 + H_2O + \frac{1}{2}O_2$$
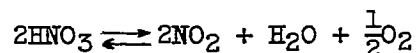
In sealed glass containers, when the vapor space is small compared to the liquid volume, this decomposition can result in pressures of the order of 1500 pounds per square inch at temperatures ranging from 160° to 250° F (reference 1). These high pressures are apparently due to

the liberated oxygen which is substantially insoluble in the liquid
phase (reference 2). As would be expected from the above equilibrium,
an addition of nitrogen dioxide materially reduces the decomposition
pressure (reference 1). In preloaded rocket missiles, the development
of high pressures could seriously affect the design, in that containers
could not be filled to maximum capacity or would require heavier walls
than are necessary to withstand normal working pressures.

Experiments in metal containers have shown that after a peak
pressure is developed the pressure gradually decreases (reference 3).
This decrease in pressure has been attributed to the corrosion reac-
tions which consume the liberated oxygen (reference 4).

The net results of the storage of concentrated nitric acids in
metal containers at high temperatures are attack on container walls,
deterioration of the acid, formation of undesirable sludges or precipi-
tates, and formation of relatively high pressures from decomposition.

Because the storage of fuming nitric acids presents a serious
operating problem, an investigation was undertaken at the NACA Lewis
laboratory to determine means for improving the storage properties of
this acid.

Experiments were conducted at 170° F for periods of up to 3 weeks
in closed aluminum containers with a capacity of approximately 100 milli-
liters. Pressure was measured by Bourdon tube gages, and the extent of
corrosion was estimated visually at the conclusion of each test.

The results of the investigation including the effects of several
inorganic additives on the rate of pressure rise and corrosion of
fuming nitric acids are presented herein.

APPARATUS

The apparatus consisted of a steel bomb containing an aluminum
test tube and was provided with a pressure gage and a vent valve. The
bombs were maintained at a temperature of 170° F in a water bath
equipped with heaters, stirrer, and temperature regulator.

Bomb. - A cross section of the bomb is shown in figure 1. It con-
sists of an aluminum test tube held in an outer shell of SAE 1020
carbon steel with an AISI 303 stainless steel cap. Both 2S and
61S aluminum were used for the test tube. Aluminum was selected for
the test tube because of its corrosion resistance in highly concen-
trated nitric acid and also because its softness relative to stainless
steel would permit the use of the lip of the test tube as a gasket
between the serrated surfaces of shell and cap. The use of stainless

steel and aluminum could possibly cause galvanic corrosion since the liquid phase is in contact with both metals. However, any galvanic corrosion would probably be negligible compared with the attack by the concentrated acid at 170° F.

By the use of appropriate fittings, a diaphragm equipped Bourdon tube gage and a hand valve were attached directly to the bomb. The entire assembly is shown by figure 2. The components were constructed of AISI 304 or AISI 316 stainless steel with the exception of the pressure-gage bushing which was 24ST aluminum. All threaded connections were sealed with a paste made from water glass and talc. This paste simplified the detection of leaks since it is originally white and turns dark brown in contact with the acid or its fumes. In no case were leaks detected between the stainless steel cap and the aluminum test tube.

Constant-temperature bath. - The constant-temperature bath was heated by 500-watt knife-blade heaters connected in series with an immersion-type thermoswitch which controlled the temperature at 170° ±2° F. The bombs were immersed in this bath up to the top of the outer shell, the remainder being exposed to the atmosphere.

Constant-temperature burette. - A jacketed 100-milliliter burette was used to load the bombs and was maintained at a constant temperature by circulating water from an ice bath. The volume of acid could be measured within ±0.1 milliliter.

Materials. - Commercial acid as received in aluminum drums was used without further treatment. Samples were transferred to glass containers and stored at 32° F until used. The red fuming nitric acid (RFNA) was blended by adding small amounts of white fuming nitric acid (WFNA) to an acid containing 20 percent or more nitrogen dioxide. The initial analysis of the two acids used is as follows:

	Percent by weight		
	HNO_3	NO_2	H_2O (by difference)
RFNA	77.7	18.7	3.6
WFNA	95.0	2.2	2.8

The density of each acid determined at 32° F by use of a Westphal balance was found to be 1.61 grams per milliliter and 1.54 grams per milliliter for RFNA and WFNA, respectively. The additives used to determine their effect on storage properties were potassium fluoride dihydrate ($KF \cdot 2H_2O$), 85-percent phosphoric acid (H_3PO_4), sodium fluoride (NaF), and potassium nitrate (KNO_3). All additives were c.p. grade and were used in amounts of 1 percent by weight of total acid.

PROCEDURE

Preparation of bomb. - All parts which have contact with the acid were degreased with acetone and then pickled in a 20-percent solution of nitric acid before assembly. Parts were then washed with water and acetone and air dried.

Prior to loading with acid, the assembled bombs were pressure-checked with helium for leaks. The gage, valve, fittings, and cap were removed as a unit from the bomb proper and calibrated by metering water from a standard 100-milliliter burette. The test tubes were similarly calibrated. The total volume of each bomb was approximately 115 milliliters and, in most cases, an ullage of approximately 15 milliliters at 32° F was selected. Since the total volume of the bomb was measured with an accuracy of ±1 milliliter and the error in acid measurement is negligible, the actual percent initial ullage varied by approximately ±1 percent from the desired value.

Loading of bomb. - The acid was metered into the test tubes from the constant-temperature burette at 32° F, at which temperature the ullage was determined by difference. The ullage at 170° F would be considerably smaller because of the thermal expansion of the acid.

One percent by weight of an additive was weighed to the nearest milligram and added to the acid in the test tube. The upper part of the bomb, previously assembled, was then mounted and the unit was placed in the bath which was already at the desired temperature. Convection currents set up in the liquid during the heating process were assumed to cause the concentration of additive to become uniform throughout the bomb. Experiments were run for a minimum of approximately 100 hours or until definite trends could be established. Upon the completion of a test, vapors were bled off and the bomb dismantled. The clear acid was sampled for analysis and visual observation was made of the corrosion products and the attack on the metal.

Acid analysis. - Acids were analyzed by the method of Air Force Specification 14159, except that the total acidity determination was by direct titration in water solution. The amount of nonvolatile impurities was determined by evaporating to dryness and then heating to red heat. All salts present were assumed to be converted to oxides. The oxide percentage was subtracted from the gross "water-by-difference" percentage to obtain the approximate net percentage of water. Three determinations were made on each sample and averaged.

Ignition-delay measurements. - The effect of $KF \cdot 2H_2O$ on the ignition delay of the acids both before and after storage was determined at a temperature of 70° F by the method reported in reference 5. The fuel was a 3:7 mixture by volume of o-toluidine and triethylamine.

RESULTS AND DISCUSSION

Comparison of Decomposition Pressures

The decomposition pressures obtained for RFNA and WFNA at 170° F and an initial ullage of 16 percent are compared in figure 3. Since the rates of change in specific volume for RFNA and WFNA are approximately the same, ullages will be equal at 170° F. The peak pressures recorded were 59 and 590 pounds per square inch gage for RFNA and WFNA, respectively. Similar data have been recorded in glass apparatus (references 1 and 6) and in metal containers (references 3 and 4).

The equilibrium equation can be written as follows:

$$2HNO_3 \rightleftharpoons 2NO_2 + H_2O + \frac{1}{2}O_2$$
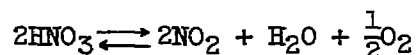
The high pressures developed with WFNA can be largely attributed to the liberated oxygen which is insoluble in highly concentrated nitric acid (reference 2). The vapor pressure of the NO_2 in WFNA is small compared with the actual pressures measured. The lower pressures developed by RFNA are primarily due to the large amount of NO_2 present, which would be expected to reverse the above equilibrium. In addition, the pressures measured with RFNA are only slightly higher than the vapor pressures reported in references 7 and 8.

The pressure developed by WFNA reaches a maximum in approximately 24 hours and then continuously decreases as shown in figure 3. The decrease in pressure can be attributed to the corrosion reaction or reactions. It has been postulated that the oxygen liberated by decomposition of the acid is consumed in oxidizing the metal container (reference 4). The exact mechanism by which this occurs is unknown, but in view of the fact that the decrease in pressure occurs after an induction period during which corrosion could start the phenomenon seems plausible. However, the increase in nitrogen dioxide and water from corrosion probably accounts for a major portion of the decrease in pressure by decreasing the equilibrium oxygen pressure. With RFNA this decrease in pressure is not as pronounced because the peak pressure is of the same order as the vapor pressure of the acid at this temperature. The data obtained in stainless steel drums also show a decrease in pressure after a 24-hour period (reference 3).

Effect of Additives on Decomposition Pressures

A comparison of the decomposition pressures of RFNA with and without potassium fluoride or phosphoric acid added is shown in figure 4. These short-term experiments, conducted at 170° F and an initial ullage

of 16 percent, indicate that both additives decrease the rate of pressure rise of this acid. Since the portions of the bombs above the top of the outer shell were not immersed in the bath, some of the scatter of data may be attributed to variation in temperature of the air surrounding this portion of the bomb.

The results of similar experiments conducted with WFNA are shown in figure 5. In this case, only the $KF \cdot 2H_2O$ decreased the rate of pressure rise. The decreasing portion of the pressure curves for the acid with H_3PO_4 and without additive can be attributed to rapid corrosion which consumes the oxygen liberated by the decomposition reaction (reference 4) and is in substantial agreement with data obtained with stainless steel drums by other investigators (reference 3).

Because, as is shown in the section Effect of Additives on Corrosiveness, the $KF \cdot 2H_2O$ appears to decrease substantially the corrosiveness of both RFNA and WFNA, it was selected for evaluation in longer-term experiments, the results of which are shown in figure 6.
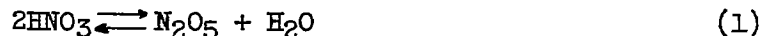
. With RFNA (fig. 6(a)), the initial rate of pressure rise (approximately first 80 hr) is similar to that shown in figure 4; however, after about 245 hours, the pressure of the acid containing $KF \cdot 2H_2O$ rises above that of the unadulterated acid and remains higher. A possible explanation of this phenomenon is that the $KF \cdot 2H_2O$ is gradually depleted by the formation of a coating on the container walls, thereby permitting decomposition to proceed and the pressure to rise. Because little corrosion occurs with acids containing $KF \cdot 2H_2O$, the oxygen formed by the decomposition reaction will not be consumed and the measured decomposition pressure will eventually exceed that of the unadulterated RFNA in which corrosion has occurred. Thus the increase in pressure for the RFNA containing $KF \cdot 2H_2O$ is due to (1) the loss of fluoride as a decomposition inhibitor and (2) the formation of a coating on the walls which prevents oxygen from reacting.

It is possible that the addition of more than 1 percent $KF \cdot 2H_2O$ or the preforming of a fluoride coating on the walls plus addition of fluoride to the acid would be more effective in reducing the rate of pressure rise and probably the peak pressure, as well.

The general increase in pressure of both systems (fig. 6(a)) after about 240 hours can be attributed to a large increase in the ambient air temperature which caused the upper portions of the bomb, which were not immersed in the bath liquid, to increase in temperature.

Results of longer-term experiments with WFNA and $KF \cdot 2H_2O$ (fig. 6(b)) are in general agreement with shorter-term storage results with regard to the rate of pressure rise. For these experiments, a cover was added to the constant-temperature bath, thereby eliminating large temperature variations and giving less scatter in pressures.

The mechanism by which $KF \cdot 2H_2O$ decreases the rate of pressure rise of nitric acid is not known, but a possible explanation is that the fluoride ion inhibits the decomposition of nitrogen pentoxide (N_2O_5) into nitrogen dioxide (NO_2) and oxygen (O_2) by the following reactions:

$$2HNO_3 \rightleftharpoons N_2O_5 + H_2O \tag{1}$$

$$N_2O_5 \longrightarrow 2NO_2 + \frac{1}{2}O_2 \tag{2}$$

$$N_2O_5 \rightleftharpoons NO_2^+ + NO_3^- \tag{3}$$

$$NO_2^+ + F^- \longrightarrow NO_2F \tag{4}$$

If nitronium fluoride (NO_2F) is not substantially dissociated, its formation by reaction (4) will tend to inhibit the decomposition reaction (2) by competing for N_2O_5 molecules. The formation of salts similar to NO_2F is described in reference 9. If the fluoride ion is gradually consumed in forming a coating on the container walls, reaction (2) would become predominant and decomposition would proceed as in the unadulterated acid.

Of the remaining two additives investigated, NaF behaved in the same manner as $KF \cdot 2H_2O$, while KNO_3 apparently had no effect on the rate of pressure rise. Since the $KF \cdot 2H_2O$ appeared more soluble in acid than the sodium salt, it was used in all subsequent experiments.

Comparison of Corrosiveness

The caps and test tubes used for short-term experiments with RFNA and WFNA with and without added $KF \cdot 2H_2O$ and H_3PO_4, are shown in figures 7 and 8, respectively. Figures 9 and 10 show the long-term effects of RFNA and WFNA with and without added $KF \cdot 2H_2O$. No attempt was made to remove the corrosion products from these parts.

From the quantity of corrosion products, RFNA (fig. 7(a)) apparently is slightly more corrosive than WFNA (fig. 8(a)). Similar results are reported in reference 10. In figures 9(a) and 10(a), it can be seen that the effects of long-term storage are similar, but more severe as would be expected. The metal surface is blackened and etched in all cases; however, no pitting of the surfaces was observed when the corrosion products were removed.

Effect of Additives on Corrosiveness

In contrast to the unadulterated acid, the absence of corrosion with acid containing $KF \cdot 2H_2O$ was very noticeable as shown in figure 7(b) and figure 8(b), for short-term experiments. In addition, the parts were not blackened or appreciably etched. The lip and the wall of the aluminum test tube were covered with a thin, white, translucent film, which was probably a fluoride coating that prevents attack by the acid. The lack of corrosion with acid containing $KF \cdot 2H_2O$ substantiates the opinion that appreciable galvanic corrosion did not occur in the bombs, since it should have occurred even in the presence of a fluoride film.

Sodium fluoride was equally as effective as the potassium salt in reducing corrosion.

The results of long-term storage of acids containing $KF \cdot 2H_2O$ are shown in figures 9(b) and 10(b). From these results, $KF \cdot 2H_2O$ is apparently less effective in reducing corrosion of stainless steel than aluminum, since the caps are appreciably etched.

The corrosion-inhibiting effect of H_3PO_4 seems to be more pronounced with WFNA than with RFNA; however, in neither case was the improvement appreciable (figs. 7(c) and 8(c)).

Potassium nitrate also did not improve the corrosion properties of nitric acid as observed visually.

Effects of Additives on Acid Deterioration and

Ignition Properties

The effects of short-term storage on acid deterioration are shown in table I for acids containing $KF \cdot 2H_2O$ and H_3PO_4 and for acids without an additive. The values shown in table I are averages for two similar experiments.

On the basis of HNO_3 and NO_2 contents, the $KF \cdot 2H_2O$ appears more effective than H_3PO_4 in decreasing acid deterioration for short-term storage and also appears to inhibit deterioration for long-term storage. Although deterioration is inhibited by $KF \cdot 2H_2O$, the consumption of oxygen by the corrosion process is also inhibited, so that pressure build-up is not necessarily reduced, as was explained previously.

The change in net water content of acids with additives appears anomalous. If the water contents are arranged in the order of decreasing corrosiveness of the acid, the acid of lowest corrosiveness has the highest water content as shown in the following table:

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Additives	Water content (percent by weight)	
	RFNA	WFNA
Before storage: None	3.5	2.7
After storage: None H_3PO_4 $KF \cdot 2H_2O$	2.0 4.3 4.2	2.4 3.6 4.4

The anomaly can be explained by the assumption that some of the water formed by decomposition and corrosion is consumed as water of hydration of the solid corrosion products formed in storage.

The effects of long-term storage on acid deterioration are shown in table II. The analysis of the acids containing $KF \cdot 2H_2O$ is probably affected to some extent by the large quantity of metallic salts present. In the case of fluorides, these salts were apparently more soluble, because no precipitates were formed. Deterioration of the acids is considerably more severe; however, $KF \cdot 2H_2O$ still appears to inhibit deterioration.

The large metallic content of the acid containing $KF \cdot 2H_2O$ can be accounted for, in part, by the nonvolatile potassium salt added. In general, the difference in nonvolatiles present in RFNA and WFNA is probably due to the difference in the solubility of corrosion products in the two acids.

Ignition delays of acids containing $KF \cdot 2H_2O$ are compared with those of acids without additives in table III. At 70° F, with the fuel used, there appears to be no effect of additive on ignition properties either before or after storage for approximately 100 hours. The variations in the data in table III are within the reproducibility of the experiment.

SUMMARY OF RESULTS

An investigation of the effects of several additives on the storage properties of fuming nitric acids contained in closed bombs having aluminum bodies and stainless steel caps was conducted at 170° F. The results of this investigation can be summarized as follows:

1. The peak decomposition pressure of white fuming nitric acid is greater by an order of magnitude than that of red fuming nitric acid at the same ullage.

2. Of the several additives studied, namely, potassium fluoride dihydrate, sodium fluoride, 85-percent phosphoric acid, and potassium nitrate, the potassium fluoride dihydrate was found to be most effective in reducing the rate of pressure rise and the corrosiveness of fuming nitric acids.

3. Acids containing potassium fluoride dihydrate suffered less deterioration than any of the other compositions studied.

4. Presence of fluoride did not affect the ignition delay at 70° F of red fuming nitric acid or white fuming nitric acid with a fuel blend of o-toluidine and triethylamine.

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TABLE I - EFFECT OF SHORT-TERM STORAGE ON

ACID COMPOSITION

	Component (Percent by weight)				
	HNO_3	NO_2	Water plus nonvolatiles	Nonvolatiles	Corrected water
RFNA					
Before storage:					
Without additive	77.7	18.7	3.6	0.07	3.5
After storage:					
Without additive	70.9	26.6	2.5	.47	2.0
Plus 1 percent $KF \cdot 2H_2O$	75.0	19.4	5.6	1.45	4.2
Plus 1 percent H_3PO_4	72.4	23.1	4.5	.21	4.3
WFNA					
Before storage:					
Without additive	95.0	2.2	2.8	0.1	2.7
After storage:					
Without additive	86.4	10.2	3.4	.97	2.4
Plus 1 percent $KF \cdot 2H_2O$	88.8	5.3	5.9	1.47	4.4
Plus 1 percent H_3PO_4	89.0	6.8	4.2	.61	3.6

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TABLE II – EFFECT OF LONG-TERM STORAGE ON

ACID COMPOSITION

	Component (Percent by weight)				
	HNO_3	NO_2	Water plus nonvolatiles	Nonvolatiles	Corrected water
RFNA					
Before storage:					
Without additive	77.7	18.7	3.6	0.07	3.5
After storage:					
Without additive	37.6	37.0	25.4	2.3	23.1
Plus 1 percent $KF \cdot 2H_2O$	53.3	28.5	18.2	9.1	9.1
WFNA					
Before storage:					
Without additive	95.0	2.2	2.8	0.1	2.7
After storage:					
Without additive	76.9	14.7	8.4	.7	7.7
Plus 1 percent $KF \cdot 2H_2O$	69.3	13.6	17.1	8.5	8.6

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TABLE III - EFFECT OF ADDED FLUORIDE ON IGNITION DELAY

OF RFNA AND WFNA AT 70° F

[Fuel: 30 percent by volume o-toluidine plus 70 per-
cent by volume triethylamine]

Acid	Ignition delay before storage (millisec)	Ignition delay after approximately 100-hour storage (millisec)
RFNA	25.5	23.0
RFNA plus 1 percent KF·2H$_2$O	20.0	20.0
WFNA	27.5	24.5
WFNA plus 1 percent KF·2H$_2$O	18.0	20.0
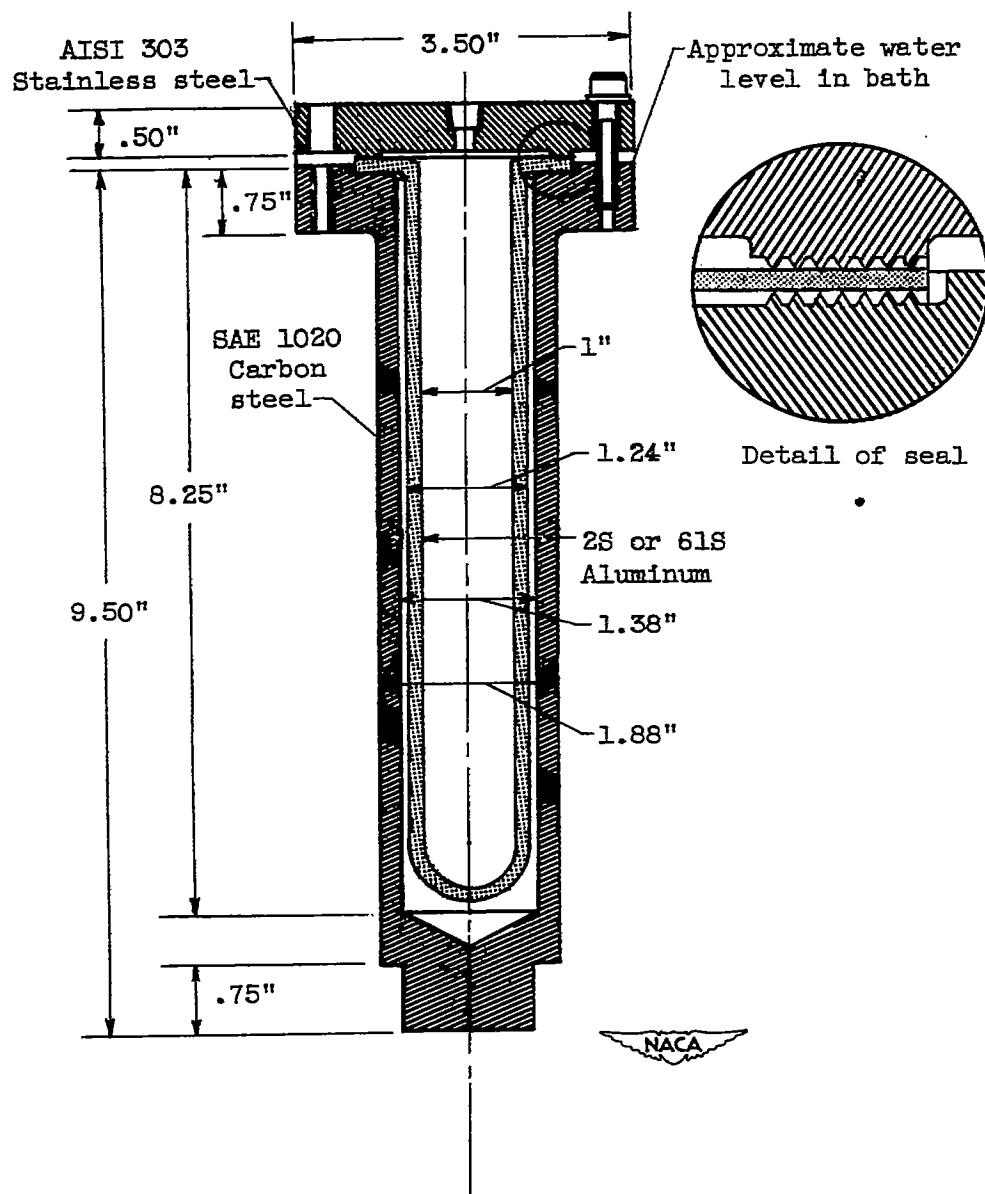
Figure 1. - Detail of storage bomb construction.

(a) Assembled view.

Vent valve

Cap

Outer shell

Separation diaphragm housing

Aluminum test cube

(b) Exploded view.

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C-30450
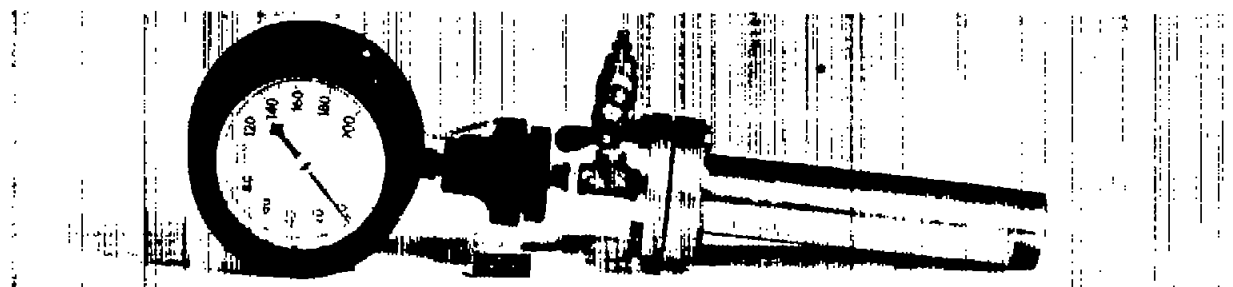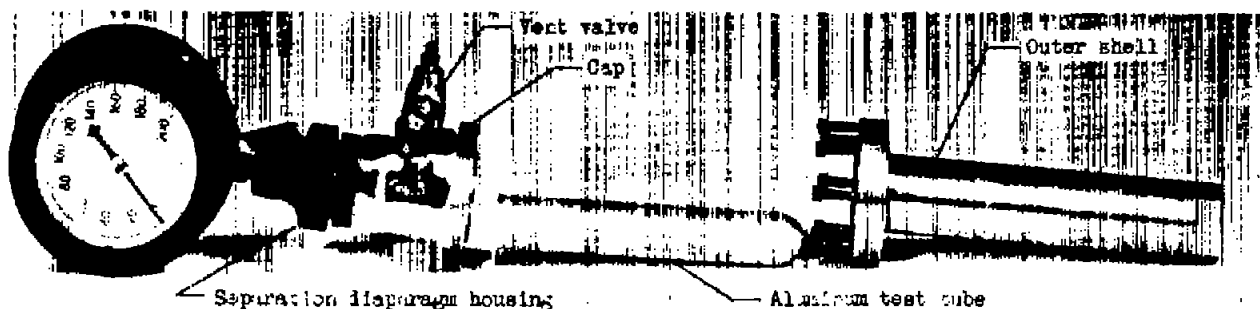
Figure 2. - Storage bomb.

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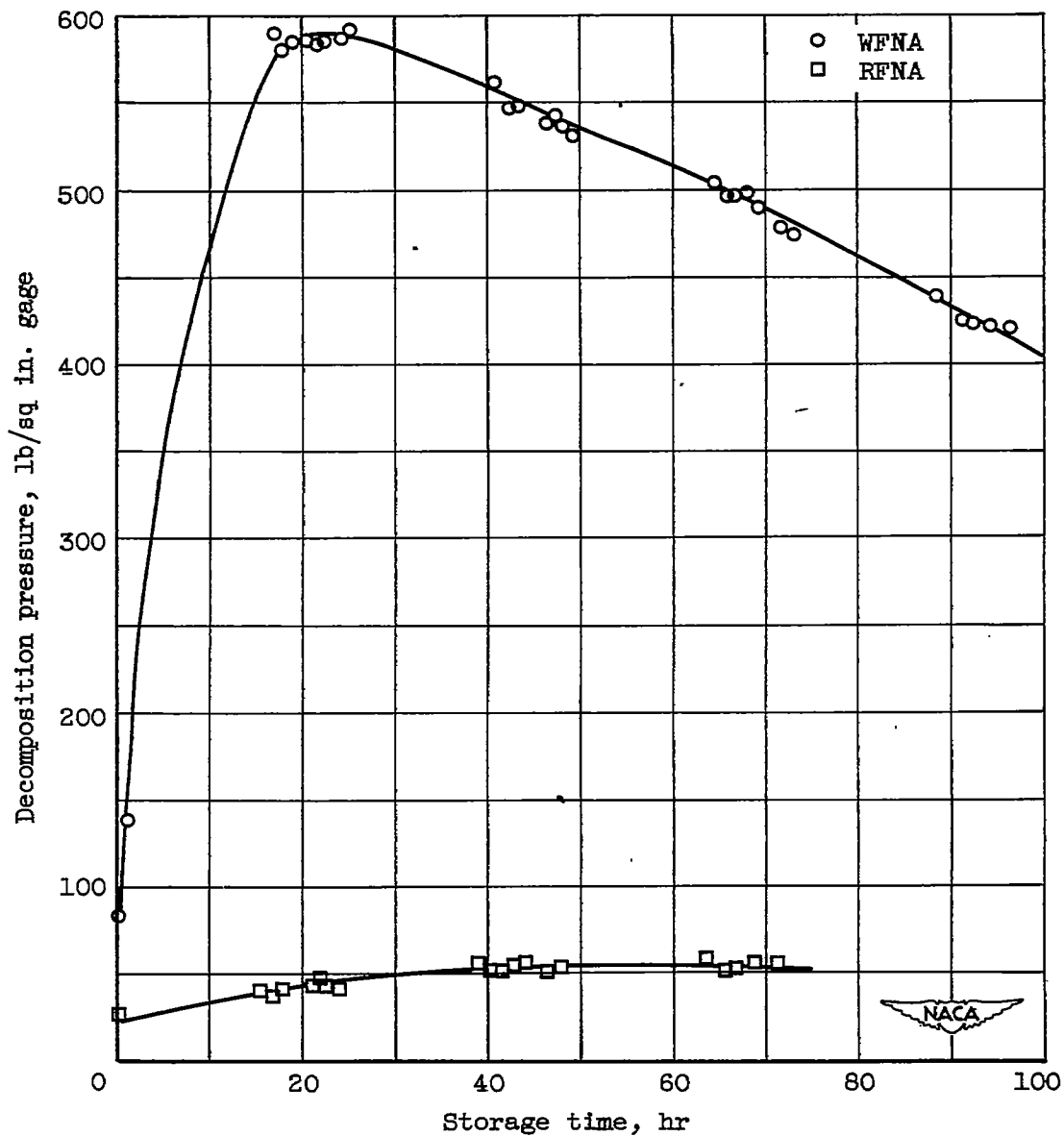
Figure 3. - Comparison of decomposition pressures of white
  fuming nitric acid (WFNA) and red fuming nitric acid
  (RFNA) at 170° F and 16 percent initial ullage.
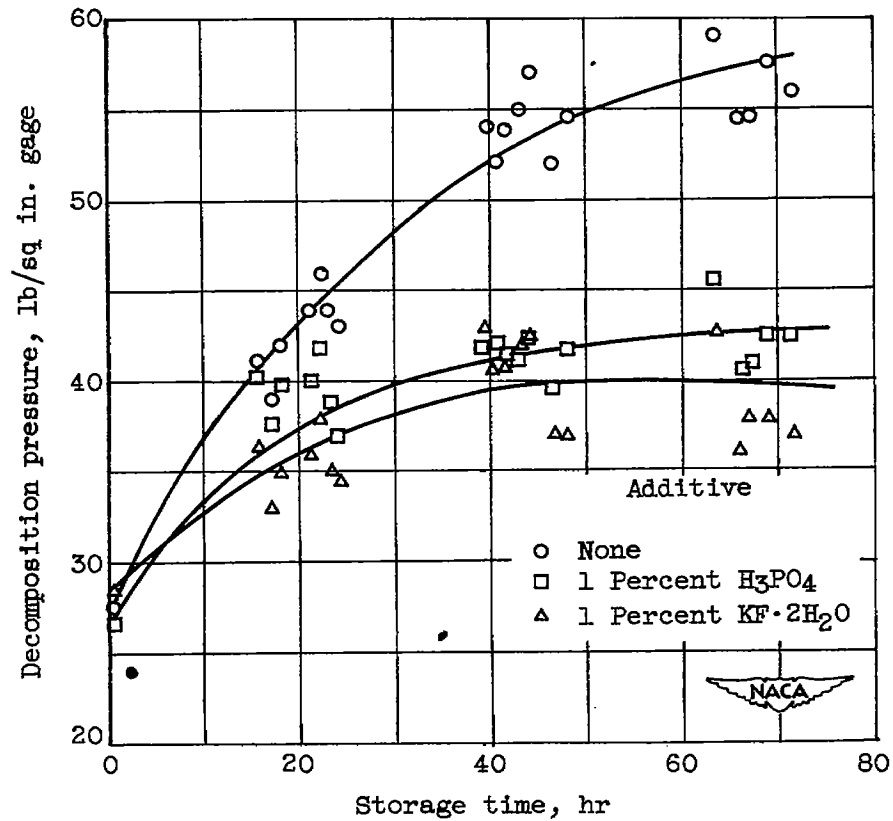
Figure 4. - Decomposition pressures of red fuming
    nitric acid and red fuming nitric acid with
    additives at 170° F and 16 percent initial
    ullage.

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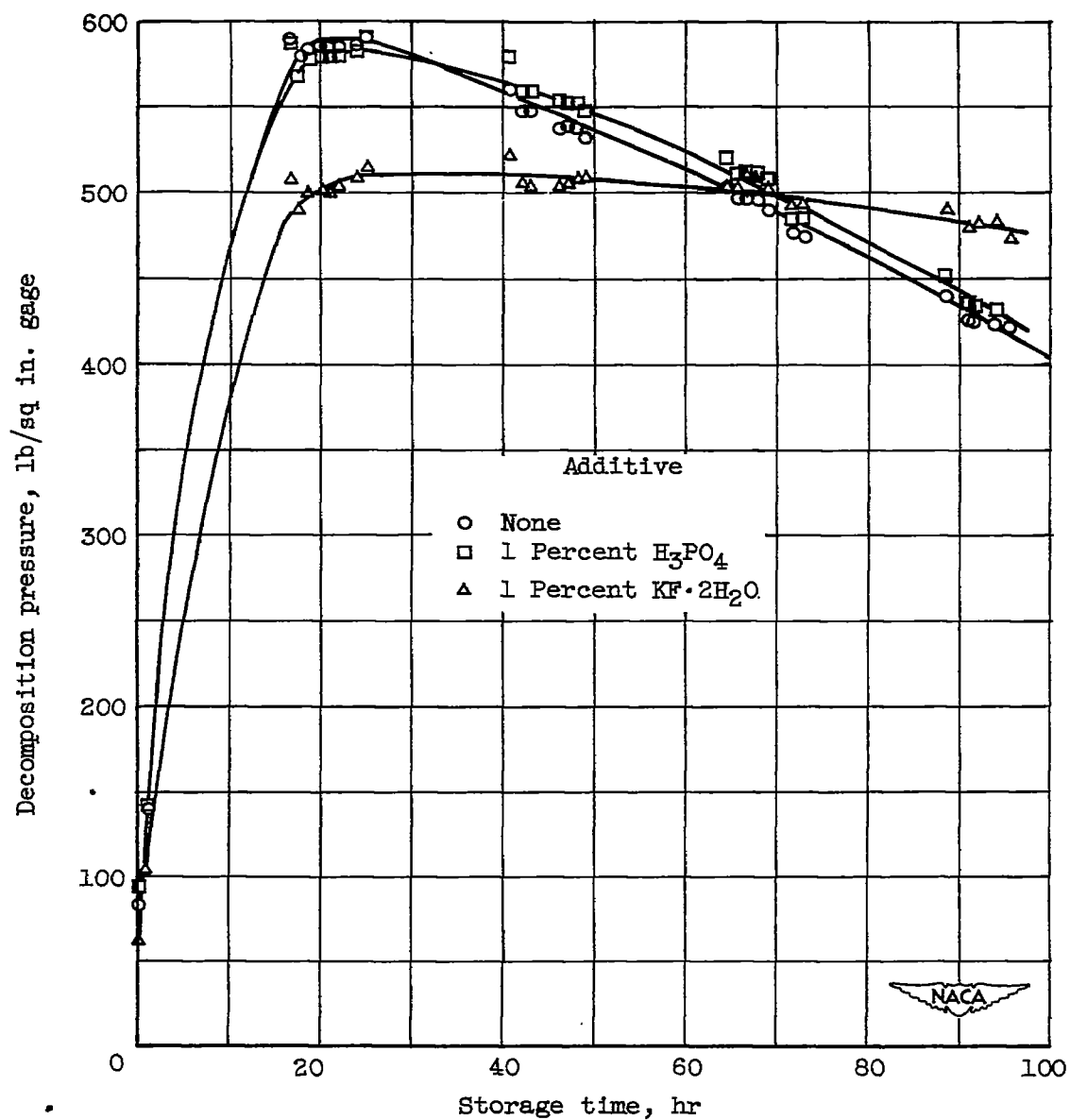
Figure 5. - Decomposition pressures of white fuming nitric acid
and white fuming nitric acid with additives at 170° F and
16 percent initial ullage.
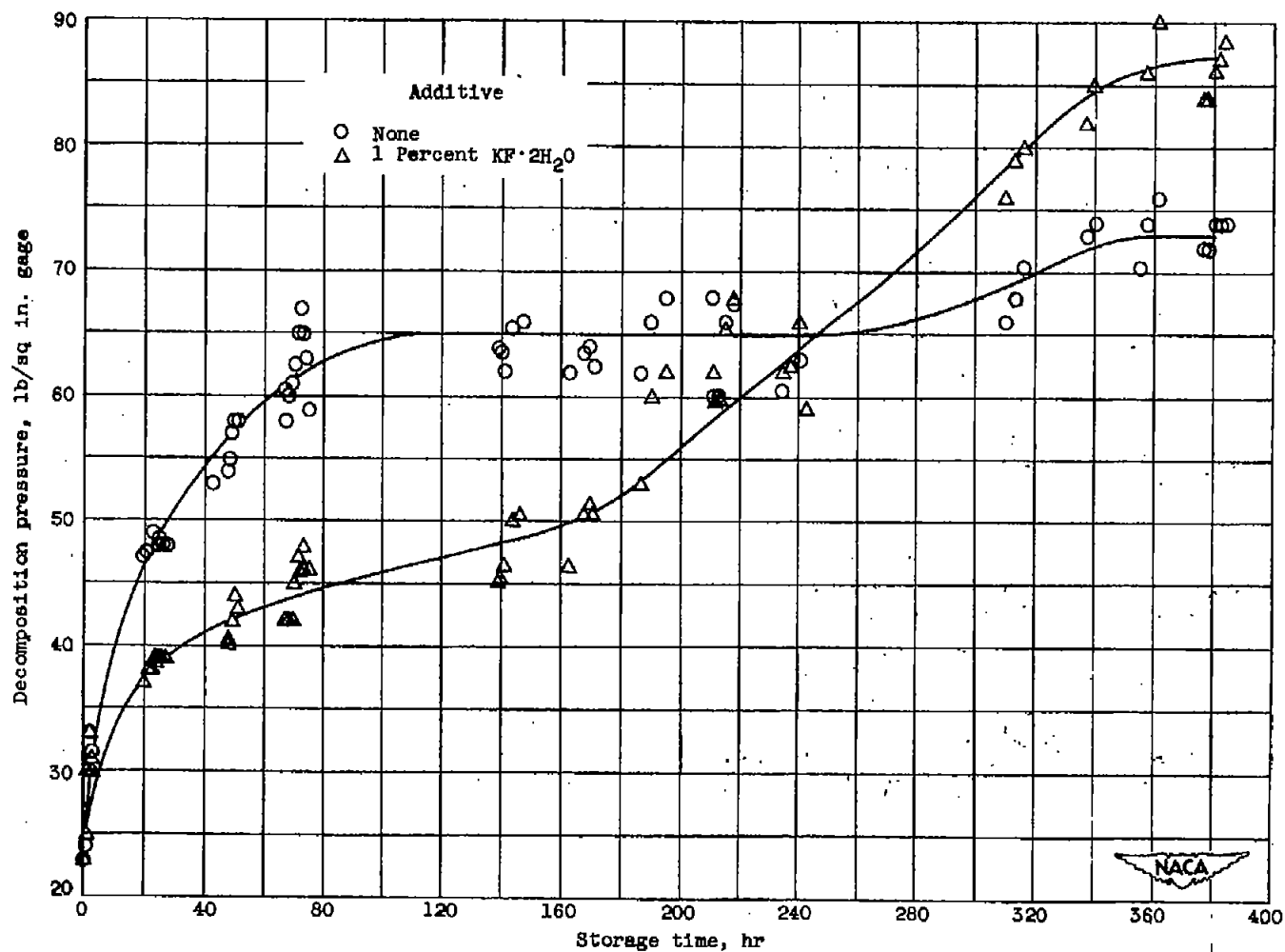
(a) Red fuming nitric acid. Initial ullage, 14 percent.

Figure 6. - Long-term decomposition pressures at 170° F.
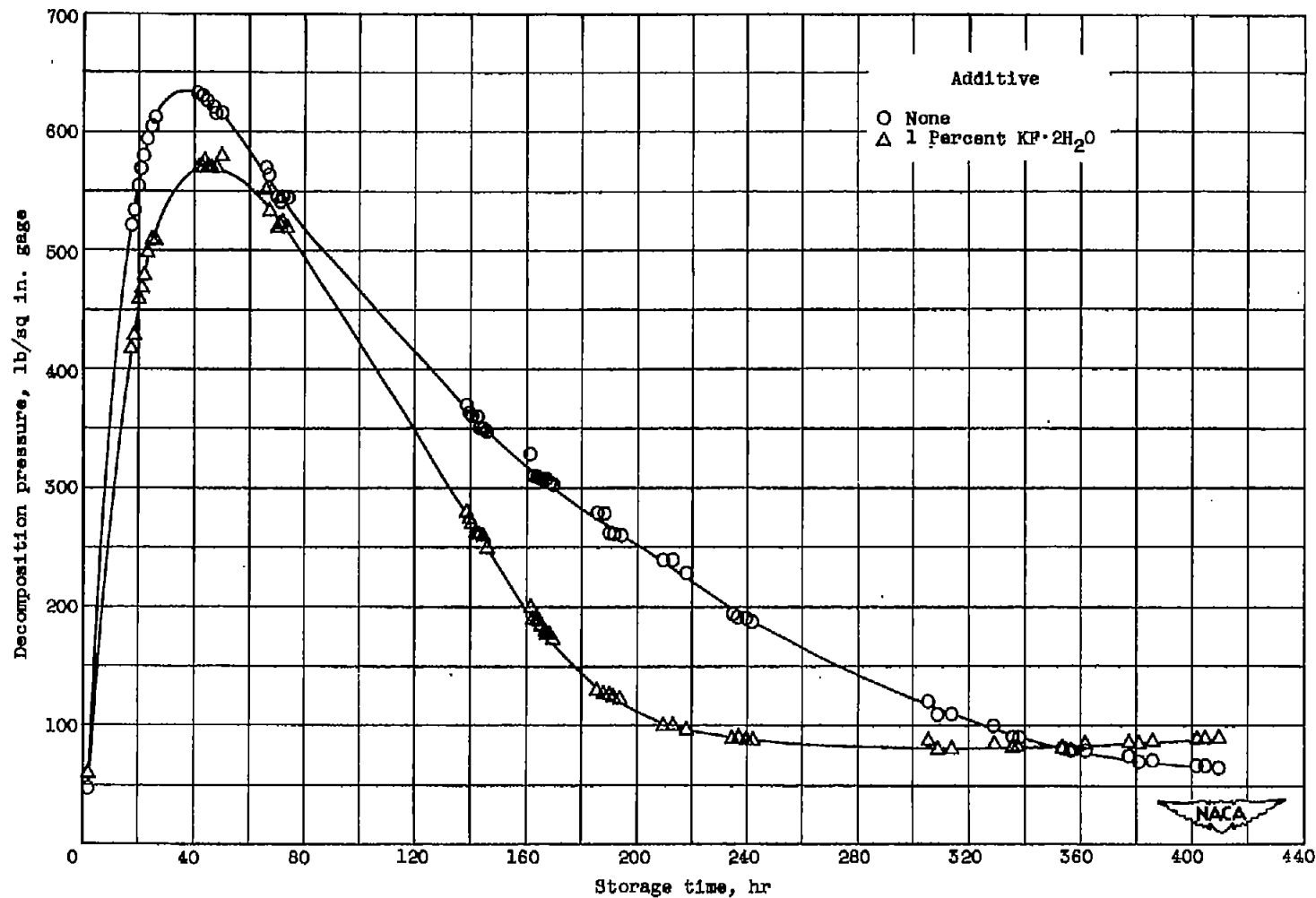
(b) White fuming nitric acid. Initial ullage, 16 percent.

Figure 6. - Concluded. Long-term decomposition pressures at 170° F.
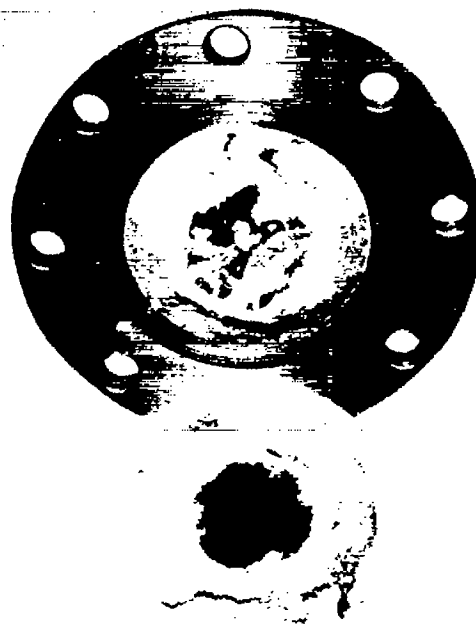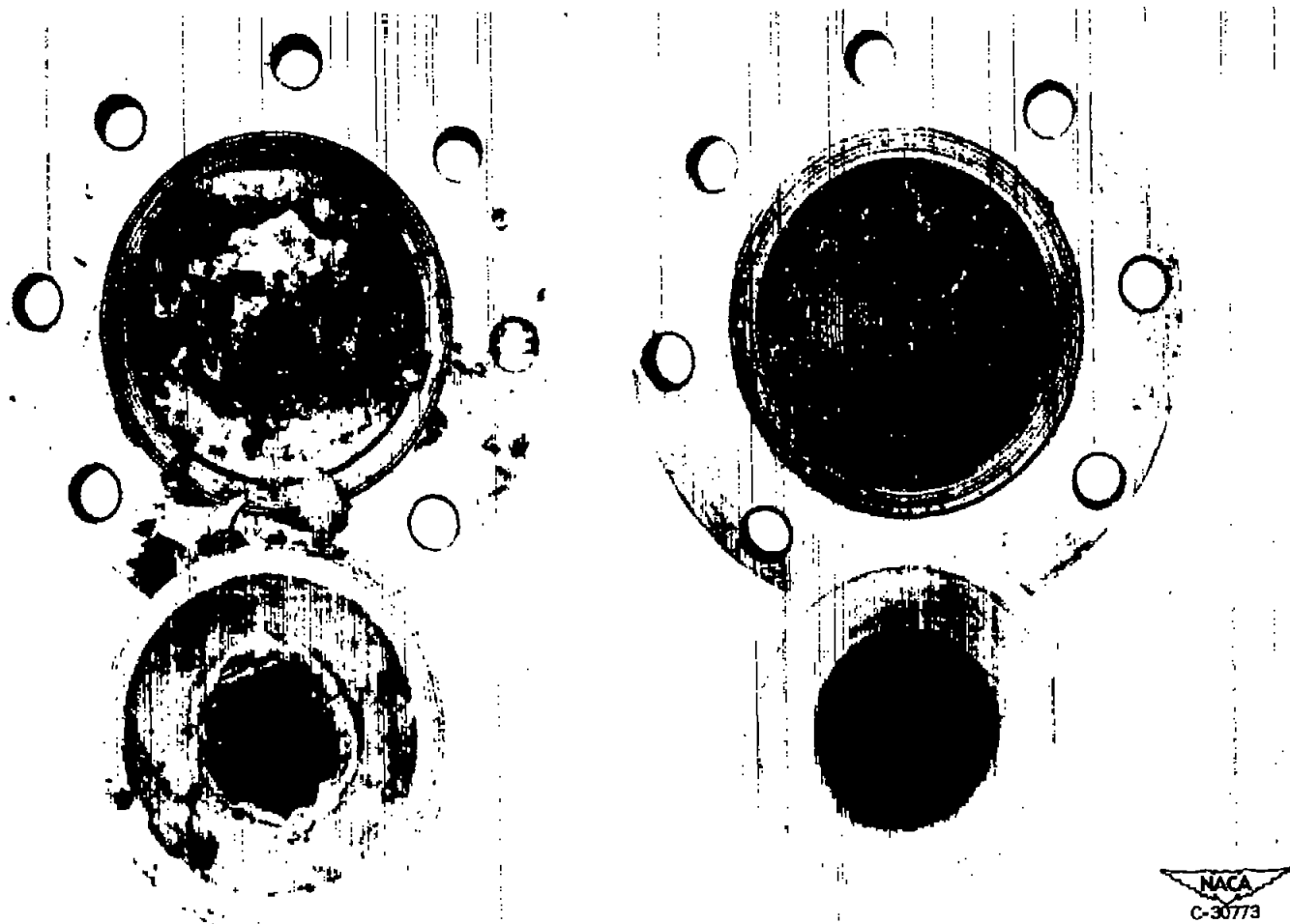
(a) Without additive.
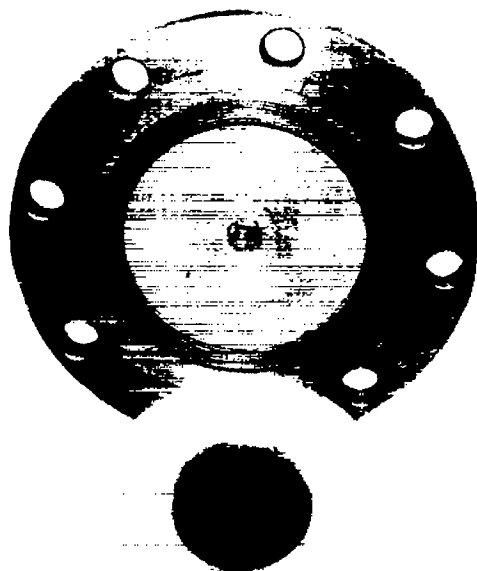
(b) Addition of 1 percent potassium
flouride dihydrate.

(c) Addition of 1 percent
phosphoric acid.

Figure 7. - Effects of additives on corrosion of stainless steel caps and aluminum test
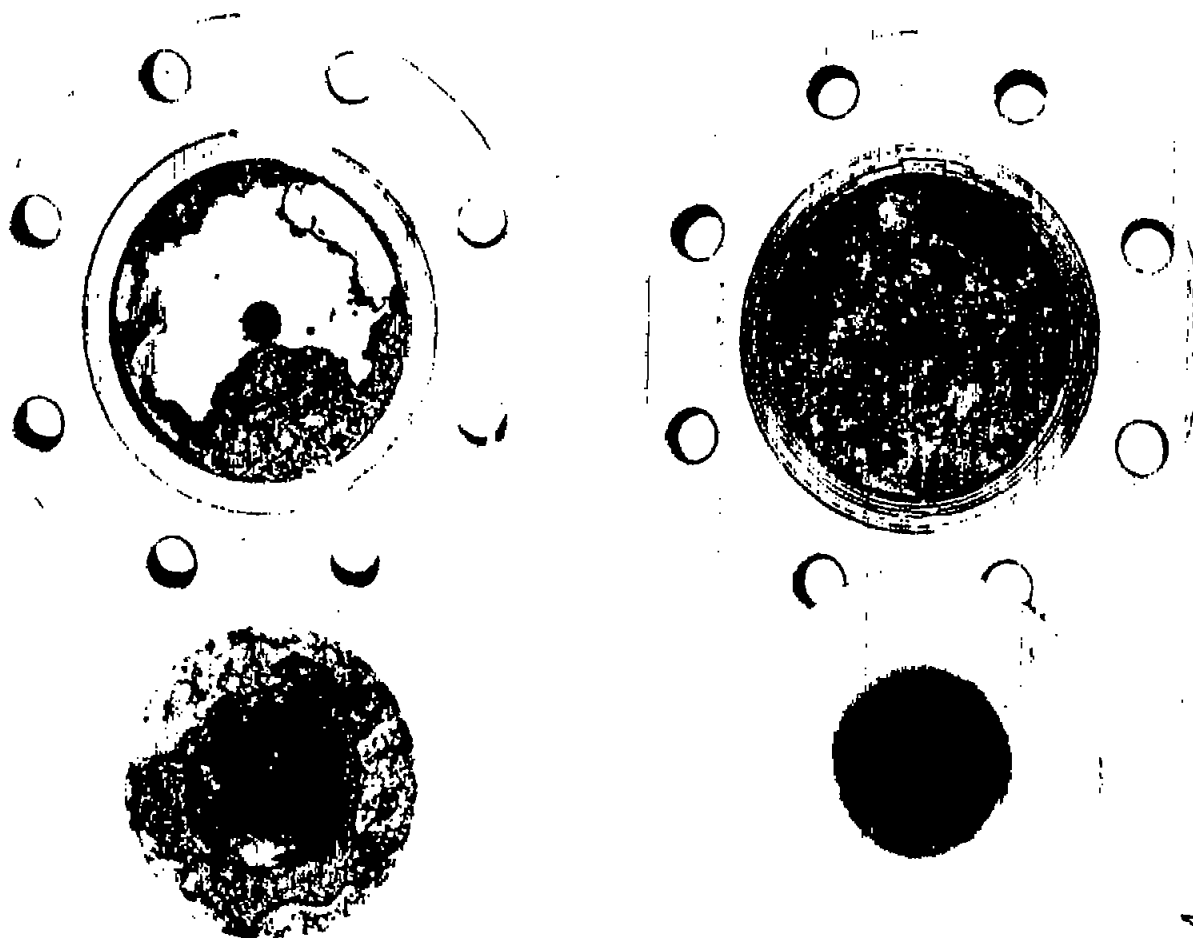tubes after approximately 100-hour storage of red fuming nitric acid at 170° F.

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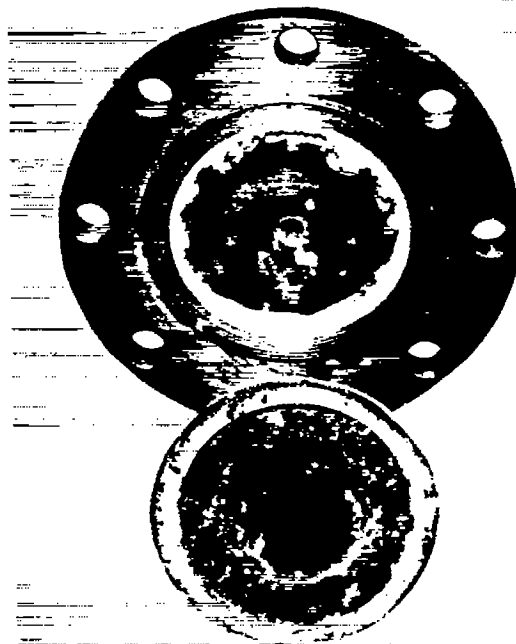
(a) Without additive.
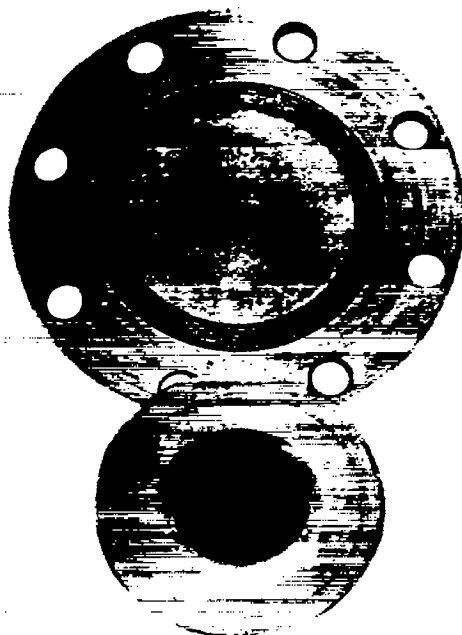
(b) Addition of 1 percent potassium
fluoride dihydrate.
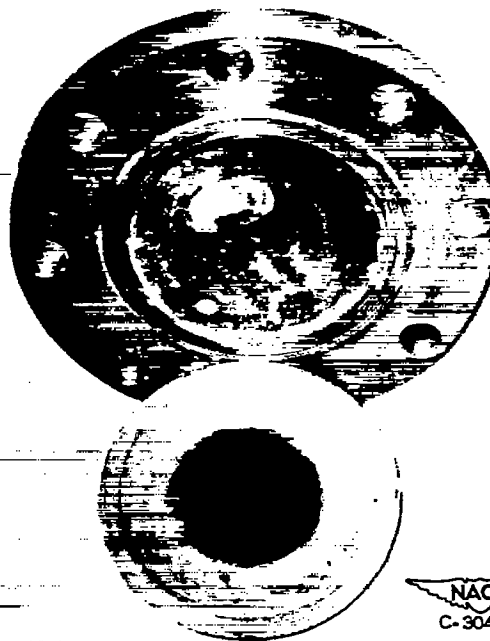
(c) Addition of 1 percent
phosphoric acid.

Figure 8. - Effects of additives on corrosion of stainless steel caps and aluminum test
tubes after approximately 100-hour storage of white fuming nitric acid at 170° F.

(a) Without additive.

(b) Addition of one percent KF·2H$_2$O.

Figure 9. - Effects of additives on corrosion of stainless steel caps and aluminum test tubes after approximately 400-hour storage of red fuming nitric acid at 170° F.
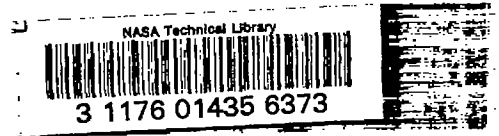
(a) Without additive.

(b) Addition of one percent KF·2H₂O.

Figure 10. - Effects of additives on corrosion of stainless steel caps and aluminum test tubes after approximately 400-hour storage of white fuming nitric acid at 170° F.